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U. S. Army
Chemical Warfare Laboratories
Technical Report

CWLR 2346

Physical Constants of Thirteen V Agents (U)

by

P. B. Coulter
J. J. Callahan
R. S. Link

FC

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December 1959 CWLR 2346

PHYSICAL CONSTANTS OF THIRTEEN V AGENTS (U)

by

P. B. Coulter
J. J. Callahan
R. S. Link

Physicochemical Research Division

Recommending Approval:

Carl M Herget

CARL M. HERGET, Ph.D.
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U. S. ARMY
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Army Chemical Center, Maryland

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(U)

FOREWORD

The object of the work described in this report was to determine certain physical constants of V agents. The work was done during the period of 5 May 1954 to 30 September 1956 and was conducted under Subproject 4-08-03-016-10, Agent Physicochemical Research (U) (formerly subprojects 4-08-03-016-01, 4-08-03-016-02, 4-08-03-016-04, and 4-08-03-016-11. Experimental data are contained in notebooks 4278, 4298, 4483, 4612, 4616, 4668, 4671, and 4708. This report was submitted for publication in May 1959.

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(C) DIGEST

(U) Physical constants have been determined on several samples of V agents synthesized in these Laboratories.

(C) The values of physical constants for VE, VG, VS, VX, EA 1728, and EA 1763 which were found are probably the same as the values which will be obtained with 100% pure samples of these compounds. Repeated syntheses and varied purifications have produced V agents of higher purities. The values shown for VP, EA 1521, EA 1576, EA 1622, VM, EA 1694, and EA 1699 are probably somewhat different from those which will be obtained with 100% pure samples.

(U)

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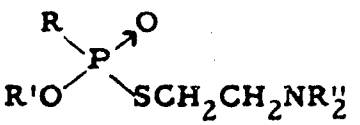
PHYSICAL CONSTANTS OF THIRTEEN V AGENTS (U)

I. (S) INTRODUCTION.

(C) Continuing research is conducted to find chemical warfare agents more effective than the present standard agents. Among the improvements sought are (a) increased toxicity or vesicancy, (b) greater stability, (c) easier penetration of protective devices, (d) increased difficulty of detection and decontamination, and (e) greater efficiency in dissemination. Research in these Laboratories and in industrial, academic, and foreign laboratories has developed compounds with greater percutaneous toxicity than GB. Among such compounds are those described in this report.

(S) The final steps of the various methods used in the preparation of V agents of the general type I,



(I)

where R, R', and R'' are the same or different alkyl groups, have been reported previously[1] and are illustrated by the following reactions:

$$RP(O)(OR')Cl + NaSCH_2CH_2NR''_2 \rightarrow I \quad (1)$$

$$[RP(O)(OR')S]\,Na + ClCH_2CH_2NR''_2 \rightarrow I \quad (2)$$

$$RP(OR')OCH_2CH_2NR''_2 + S \rightarrow RP(OR')(S)OCH_2CH_2NR''_2 \xrightarrow{\Delta} I \quad (3)$$

$$RP(S)(OR')Cl + HOCH_2CH_2\,NR''_2 \rightarrow RP(OR')(S)OCH_2CH_2NR''_2 \xrightarrow{\Delta} I \quad (4)$$

(C) Compounds VP (EA 1511) and EA 1576, while V agents, do not have the characteristic structure of type I and are not prepared by the above syntheses.

II. (C) EXPERIMENTAL.

A. (U) Materials.

The samples of the V agents used in the work reported here were synthesized in these Laboratories, and no attempts for additional purification were made by the authors. Analytical data received with the samples are shown in table 1, appendix.

B. (U) Equipment and Procedures.

Equipment and procedures for the measurement of density, viscosity, surface tension, and freezing point have been previously described.[2, 3] The measurements are precise to ±0.02%, ±0.5%, ±0.4%, and ±0.01°C, respectively. Some surface-tension values reported here were determined by either the capillary-rise method, the DuNouy ring method, or the Cassel tensiometer (National Instrument Laboratories, Riverdale, Md.). The latter is essentially a bubble-pressure tensiometer, which requires only a few drops of liquid. Water solubility was measured by the cloud-point method in a modified Beckmann freezing-point apparatus. Flash points were determined in a semimicro apparatus similar to that reported by McCutchan and Young.[4]

C. (C) Results.

Physical properties of 13 V agents are shown in the table below. The values for VE, VG, VS, and VX are from the samples considered best. The single samples of EA 1728 and EA 1763 gave consolute curves in water that indicated high purity. Since repeated syntheses and varied purifications have produced V agents of increased purity, it is probable that the physical constants of VP, EA 1521, EA 1576, EA 1622, VM, EA 1694, and EA 1699 may be subsequently changed. Detailed results are given in the appendix.

III. (S) DISCUSSION.

(S) Attempts were made to observe correlation among the physical properties of the 13 V agents covered in this report. Trends were apparent in the phosphonothioates VE, EA 1622, VM, VS, EA 1694, EA 1699, VX, EA 1728, and EA 1763, but considerable deviation from the trends were noted if gross changes were made in the substituents on the phosphorus, e.g., VG, VP, EA 1521, and EA 1576.

(C) Table

Physical Properties of V Agents (U)

Agent	Density at 25°C*	Viscosity at 25°C*	Surface tension at 25°C*	Parachor		Melting point	Flash point**	Lower consolute temp in water
				Calculated	Found			
	g/ml	cs	dynes/cm				C°	
VG (EA 1508)	1.0457	4.74	31.0	617.6	607.8	ca.-51	-	25.5
VP (EA 1511)	1.023	29.6	30.4	678.2	682.5	-	-	-
VE (EA 1517)	1.0180	5.44	29.5	597.6	587.7	-	157	41.4
EA 1521	0.995	-	-	807.6	-	-	-	-
EA 1576	1.0829	23.3	32.4	686.9	670.5	-	-	-
EA 1622	1.023	6.10	29.7	594.6	578.1	-	135	-
VM (EA 1664)	1.0311	5.85	31.2	557.6	548.6	-	-	74
VS (EA 1677)	1.0016	9.36	29.9	671.6	657.0	ca.-35	168	ca.-5
EA 1694	1.0453	4.92	31.5	517.6	510.6	-	-	misc. 0 to 100
EA 1699	1.0600	5.31	32.0	477.6	474.1	-	-	-
VX (EA 1701)	1.0083	9.96	31.6	631.6	628.7	-	159	9.4
EA 1728	0.9899	11.4	29.2	668.6	660.8	ca.-12	170	-1.6
EA 1763	0.9973	11.3	30.2	671.6	661.5	-	-	below 0

* (U) Values at other temperatures are contained in the appendix.

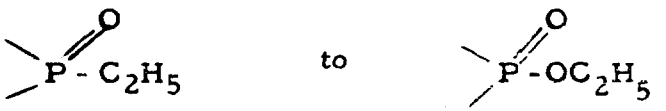
** (U) Values determined in a semimicro apparatus.

(S) Conclusions regarding the trends were based on those samples of each compound which were considered the best of all received. The principal criteria of the best samples were the water-solubility curves. The critical solution temperature of a tertiary amine is a very sensitive indicator of both water-soluble and water-insoluble impurties. The shape of the curve of cloud-point temperature versus concentration of amine in water gives not only a qualitative estimate of the ratio of water soluble to water-insoluble impurities but also can give an estimate of total impurities.[5,6] Because the samples for which cloud points were measured were prepared by essentially the same method, the same types of impurities would be expected.

(S) Where several samples of a compound were received, an increase in purity (based on water-solubility curves) was usually noted in the later samples. In view of this observation, it was considered probable that the single samples of EA 1622, EA 1694, and EA 1699 were of relatively low purity. With these qualifications in mind, the following conclusions were made:

1. (C) Density.

The change in density resulting from changes in R, R', and R'' in the structure $RP(O)(OR')S\text{-}C_2N_4NR''_2$ was nearly a decreasing linear function of total carbon atoms or of molecular weight. Within the limit of sensitivity shown by density determinations, and considering the small number of compounds examined, it appeared that little difference in density resulted from substitution at a specific site in the molecule. It is interesting to note that changing



(VE to VG) is accompanied by an increase in density. Good agreement with the United Kingdom density values[7] was observed when samples were of comparable purity.

2. (U) Surface Tension.

Parachor for each of the compounds was calculated using the atomic and structural values of Mumford and Phillips.[8] The calculated parachors were then compared with parachors obtained by substitution of surface tension in the expression:

where

$$P = \alpha^{1/4} M/D$$

P = parachor

α = surface tension at 25°C, dynes/cm

M = formula weight

D = density at 25°C, g/ml

Parachors for pure homologs of a series should be an additive function. The magnitude of deviation between calculated and measured parachors for some of the compounds reported here has indicated the presence of impurities.

3. (S) Viscosity.

Plots of $\log_{10}$ viscosity versus molecular weight or total carbon atoms both gave the same type curves. The decrease in $\log_{10}$ viscosity from increased size of R (from $-CH_3$ to $-C_2H_5$) was the same (EA 1699 to EA 1694, VM to VE, and VX to VS). The increase of log viscosity from changed $(R'')_2$ [from $-CH_3$ to $-C_2H_5$ to $-CH(CH_3)_2$] was the same (EA 1699 to VM to VX and EA 1694 to VE to VS). These changes suggest a limited potential for the tailoring of viscosity to a desired value.

4. (C) Flash Point.

Flash points of five V agents (VE, EA 1622, VS, VX, and EA 1728) were measured with a few drops of each of the liquids. All of the compounds flashed in the temperature range from 135° to 170°C. Flash points of a homologous series of organic liquids are usually a function of vapor pressure, or of total carbon atoms in the molecule, and are also affected by the position of the carbon atoms in branched molecules. Relatively pure samples of VS and EA 1728, each with 12 carbon atoms, had flash points of 168° and 170°C, respectively. Larger differences as

shown by the 10-carbon atom EA 1622 (flash point of 135°C) and EA 1728 may have been caused by structural differences but may also have been caused by the presence of relatively volatile impurities in EA 1622.

5. (S) Melting Points.

Melting points were obtained with only three of the compounds discussed in this report: VG, VS, and EA 1728. VG with an ethoxy group substituted for R in the structure $RP(O)(OR')SC_2H_4NR''_2$ had a melting point of ca. -51°C. VS with R = $-C_2H_5$, R' = $-C_2H_5$, and R'' = $-CH(CH_3)_2$ had a melting point of ca. -35°C. EA 1728 with R = $-CH_3$, R' = R'' = $-CH(CH_3)_2$ had a melting point of ca. -12°C. Predictions based on these few melting points are subject to considerable error, but it is considered probable that all of the compounds having the above structure and with R, R' and R'' being any combination of methyl, ethyl, and isopropyl will freeze below -12°C, except for the single compound which has R = R' = R'' = $-CH(CH_3)_2$. This compound has not been synthesized, but its melting point is probably above -12°C.

6. (C) Lower Consolute Temperature.

It is apparent that the lower consolute temperatures of VE, VM, VS, EA 1694, VX, EA 1728, and EA 1763 in water are a decreasing function of total carbon atoms in the molecule. Sufficient data have not yet been obtained to permit strict conclusions concerning the effect of carbon-atom position in the molecule upon lower consolute temperature. However, there were promising indications that such a relation existed, and it is hoped that the relation will be disclosed with the aid of more sensitive apparatus (presently being assembled) and with samples of high purity.

7. (C) Vapor Pressure.

Only limited vapor-pressure data have been obtained to date. Distillation data and other information[7,9] show the V agents to have vapor pressures in the 10^{-3} mm Hg range at normal temperatures. A Knudsen effusion cell has been fabricated, and calibration runs with standard samples having vapor pressures in the range from 10^{-5} to 10^{-3} mm Hg are being made. A micromanometer or differential diaphragm gage is also being tested with materials having vapor pressures in the range from 10^{-3} to 1 mm Hg. Subsequent reports will include vapor-pressure data of agents and candidate agents as the data are obtained.

IV. (C) CONCLUSIONS.

The values of physical constants for VE, VG, VS, VX, EA 1728, and EA 1763 which were found are probably the same as the values which will be obtained with 100% pure samples of these compounds. Repeated syntheses and varied purifications have produced V agents of higher purities. The values shown for VP, EA 1521, EA 1576, EA 1622, VM, EA 1694 and EA 1699 are probably somewhat different from those which will be obtained with 100% pure samples.

V. (U) LITERATURE CITED.

1. Canfield, J. H., Kagan, B., and Hoffmann, F. W. CWLR 2048. Preparation of V Agents in Aqueous Medium. August 1956.

2. Zeffert, B. M., and Coulter, P. B. TCR 36. Progress Report. Physical Constants of G Series Compounds. June 1949.

3. Zeffert, B. M., and Coulter, P. B. TDMR 1292. Physical Constants of G Series Compounds - Compounds EA 1210, EA 1211, EA 1212, EA 1213, EA 1214. July 1947.

4. McCutchan, P., and Young, D. A. Anal. Chem. 24, 1974 (1952).

5. Neale, E. PTP 470. The Solubility of Phosphorothiolates and Related Compounds. Part 1. 1955.

6. Callahan, J. J., Coulter, P. B., and Zeffert, B. M. CWLR 2074. Solubility Studies With VE and VG. October 1956.

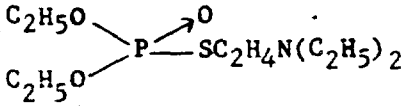
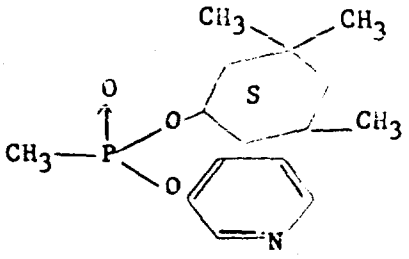
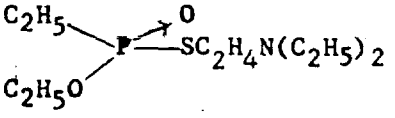
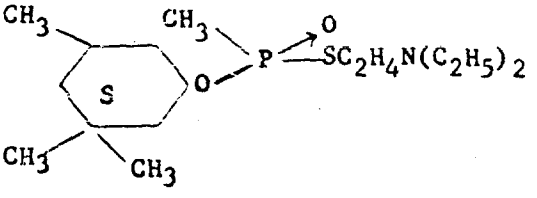
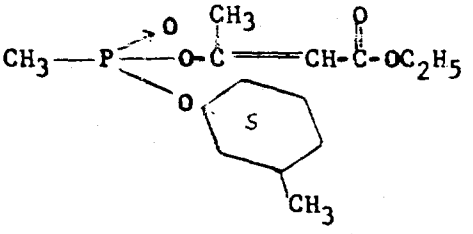
7. Tenth Tripartite Conference. ETF 500 E-8777. CW Agents. Item 4. 1955.

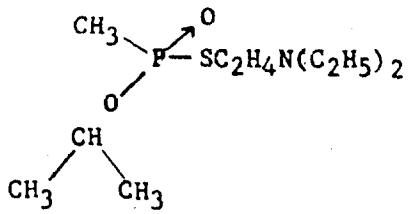
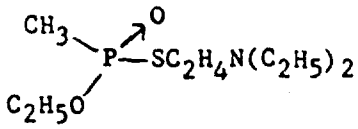
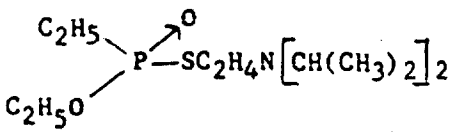
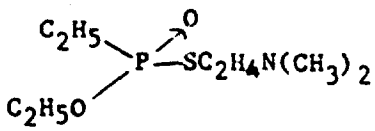
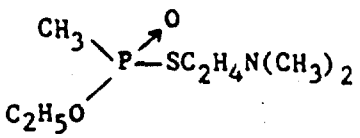
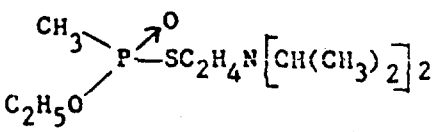
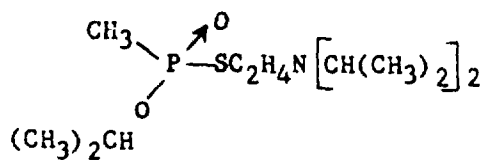
8. Mumford, S. A., and Phillips, J. W. C. J. Chem. Soc. 1929, 2112.

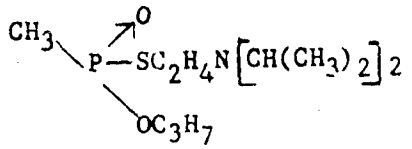
9. Trick, G. S. Suffield Technical Note No. 12. The Vapour Pressure of VE. March 1956.

(S) GLOSSARY

V agents are that class of toxic chemical agents that contain phosphorus, have high anticholinesterase activity, are highly toxic by the percutaneous route, and have a very low vapor pressure. Examples are the following compounds synthesized by these Laboratories.

Agent	Formula	Name
VG (EA 1508)		O,O-diethyl S-(2-diethylamino)ethylphosphorothioate
VP (EA 1511)		3-pyridyl 3,3,5-trimethylcyclohexyl methylphosphonate
VE (EA 1517)		O-ethyl S-(2-diethylaminoethyl) ethylphosphonothioate
EA 1521		O-3,3,5-trimethylcyclohexyl S-(2-diethylaminoethyl) methylphosphonothioate
EA 1576		2-ethoxycarbonyl-1-methylvinyl 3-methylcyclohexyl methylphosphonate

Agent	Formula	Name
EA 1622		O-isopropyl S-(2-diethylaminoethyl) methylphosphonothioate
VM (EA 1664)		O-ethyl S-(2-diethylaminoethyl) methylphosphonothioate
VS (EA 1677)		O-ethyl S-(2-diisopropylaminoethyl) ethylphosphonothioate
EA 1694		O-ethyl S-(2-dimethylaminoethyl) ethylphosphonothioate
EA 1699		O-ethyl S-(2-dimethylaminoethyl) methylphosphonothioate
VX (EA 1701)		O-ethyl S-(2-diisopropylaminoethyl) methylphosphonothioate
EA 1728		O-isopropyl S-(2-diisopropylaminoethyl) methylphosphonothioate

Agent	Formula	Name
EA 1763		O-n-propyl S-(2-diisopropyl-aminoethyl) methylphosphono-thioate

(C) APPENDIX

DETAILED RESULTS OF STUDIES WITH V AGENTS (U)

1. (C) VG (EA 1508).

(C) A total of six samples of VG were received during the period from 5 May 1954 to 18 May 1955. Analytical data for five of the samples are included in table 1. Measured physical properties of all the samples are listed in table 2. Density, viscosity, and surface tension of samples 5 May 1954, 4 June 1954, fraction 1, and 4 June 1954, fraction 4, were run before the virtues of water solubility for purity estimation had been considered.* Water-solubility curves and lower-consolute temperatures (L.C.T.) were obtained for all subsequent samples where the type and quantity of impurities permitted such measurements.**

(C) Such small amounts of a sample received on 19 April 1955 and of sample 2897 were received that measurements of any physical properties besides L.C.T. were precluded. This was unfortunate in that the sample of 19 April 1955 yielded the highest L.C.T. and was probably slightly purer than sample 2900. Only one impure sample of VG has been frozen. A melting point of -50.7°C has been observed, indicating that pure material will probably freeze somewhat above -50°C.

(C) Sample 2900 was the best sample for which several physical properties are available. Based on water-solubility data, it was considered probable that both density and viscosity of 100% VG will be only slightly greater than the values found for sample 2900. More extensive water-solubility data are given elsewhere.**

---

* (U) Neale, E. PTP 470. The Solubility of Phosphorothiolates and Related Compounds. Part 1. 1955.
Callahan, J. J., Coulter, P. B., and Zeffert, B. M. CWLR 2074. Solubility Studies With VE and VG. October 1956.

** (U) Callahan, J. J., Coulter, P. B., and Zeffert, B. M. CWLR 2074. Solubility Studies With VE and VG. October 1956.

(C)

TABLE 1

ANALYSES OF V-AGENT SAMPLES

V agent	Sample	Boiling point		Refractive index	Analytical data				
		Temperature	Pressure		C	H	P	N	S
		°C	mm Hg	n_D^{25}			%		
VG (EA 1508)	Theoretical	-	-	-	44.59	8.98	11.50	5.20	11.90
	5 May 54	-	-	1.4655 (1.4645 on 30 Apr 55	45.0	9.2	11.68	-	-
	4 Jun 54, fraction 1	98-99	0.075-0.080	1.4655	44.8	9.3	11.34	5.23	12.01
	4 Jun 54, fraction 4	94-98	0.075-0.080	1.4632	42.3	8.6	11.48	4.88	11.69
	2897	-	-	1.4647	45.6	9.2	11.43	5.48	11.90
	2900	88-89	0.13	1.4658	44.8	9.2	11.63	5.19	12.31
VP (EA 1511)	Theoretical	-	-	-	60.59	8.14	10.42	4.7	-
	2216	108	0.05	-	60.27	8.46	10.71	2.8	-
VE (EA 1517)	Theoretical	-	-	-	47.41	9.55	12.23	5.53	12.66
	23 Apr 54	66-67	0.015	-	-	-	-	-	-
	2884	80-85	0.100	1.4770	47.45	9.75	12.25	5.7	12.58
	2906	81-82	0.120	1.4780	47.5	9.7	12.42	5.50	12.47
	3100	75-78	0.020-0.045	1.4778	43.7	9.3	12.23	5.55	12.58
	PB no. 4	68-69 81-82	0.004 0.075	1.4766	-	-	-	-	-
	9003	73.5-77	0.04-0.06	1.4733	47.1	9.4	12.51	5.45	12.73
EA 1521	Theoretical	-	-	-	57.28	10.32	9.23	4.18	9.56
	2264	105-106	0.03	-	58.0	10.2	8.70	4.16	9.4
EA 1576	Theoretical	-	-	-	55.25	8.28	10.18	-	-
	2396-A	93-96	0.015	-	56.3	9.7	10.2	-	-
EA 1622	Theoretical	-	-	-	47.41	9.55	12.23	5.53	12.66
	3098	93-81	0.045-0.070	-	45.7	8.1	13.74	4.85	11.88
VH (EA 1664)	Theoretical	-	-	-	45.17	9.27	12.94	5.85	13.40
	2860-b	75-76	0.100	1.4799	45.3	9.2	12.4	6.21	13.79
VH (EA 1664) (contd.)	3051, fraction 2	75-77	0.060	1.4784	45.3	9.2	13.18	6.61	13.49
	3051, fraction 3								
	9002	-	-	-	45.1	10.4	-	5.85	13.69
	9012	-	-	-	45.1	9.3	13.24	5.74	14.14
VS (EA 1577)	Theoretical	-	-	-	51.22	10.03	11.01	4.98	11.39
	3089	80-83.5	0.005-0.010	1.4755	50.4	9.8	11.47	5.05	11.82
	9004	81-82	0.030-0.035	1.4756	51.1	9.9	11.35	4.91	11.45
	9011, fraction 12	74-80	0.030-0.070	1.4688	49.3	9.8	13.29	4.26	8.65
	9011, fraction 13	74-84	0.030-0.080	1.4745	-	-	-	-	-
EA 1594	Theoretical	-	-	-	42.65	8.95	13.75	6.22	16.23
	W	-	-	-	42.57	9.08	13.86	6.24	14.38
EA 1699	Theoretical	-	-	-	39.90	8.59	14.66	6.63	15.18
	3034	-	-	-	39.6	8.7	14.32	6.88	-
VX (EA 1701)	Theoretical	-	-	-	49.41	9.80	11.58	5.24	11.99
	3058	80-86	0.085-0.100	1.4757 1.4765	49.3	8.66	11.40	5.4 6.3	12.18
	PB no. 12	77-87	0.085-0.095	1.4773	-	-	-	-	-
	3121	77-84	0.085-0.090	1.4764 1.4774	49.7	9.9	11.96	5.3	11.88
	3132	79-83	0.085-0.040	1.4774	48.1	9.3	11.73	5.28	11.65
	9000	72-75	0.085-0.080	1.4774	49.5	9.9	11.68	5.19	12.2
	9005-3	81-82	0.080	1.4776	49.6	9.6	11.64	5.3	11.99
	9009	-	-	-	49.8	9.8	11.53	5.25	11.97
EA 1728	Theoretical	-	-	-	51.22	10.03	11.01	4.98	11.39
	9022, fraction 2	92-94	0.180-0.230	1.4738	-	-	11.0	5.0	11.32
EA 1763	Theoretical	-	-	-	51.22	10.03	11.01	4.98	11.39
	9167	-	-	-	51.5	10.1	11.26	4.97	11.31

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TABLE 2

PHYSICAL PROPERTIES OF VG (EA 1508) SAMPLES (U)

Sample	Density at various temp			Viscosity at various temp			Surface tension at 25°C *	Lower consolute temp in water
	25°C	35°C	50°C	25°C	35°C	50°C	25°C *	
	g/ml			cs			dynes/cm	°C
5 May 54	1.0439	1.0350	1.0217	4.58	3.55	2.58	30.6	-
4 Jun 54, fraction 1	1.0449	1.0362	1.0231	4.71	3.69	2.70	31.4	-
4 Jun 54, fraction 4	1.0421	1.0332	1.0201	4.37	3.43	2.49	31.1	-
19 Apr 55	-	-	-	-	-	-	-	25.5
2897	-	-	-	-	-	-	-	23.5
2900	1.0457	1.0368	1.0238	4.74	3.66	2.65	31.0	25.1

* (U) Measured by the DuNouy ring method.

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2. (C) VP (EA 1511).

(C) One sample of VP was received and the physical properties measured are listed in table 3. Frequent vitrifaction in liquid nitrogen followed by storage at temperatures from -70° to -10°C did not yield crystals.

(C) TABLE 3

PHYSICAL PROPERTIES OF VP (EA 1511) (ANAL. 2216) (U)

Temperature	Density	Viscosity	Surface tension*	Water solubility**
°C	g/ml	cs	dynes/cm	wt %
25	1.023	29.6	30.4	-
35	1.016	17.0	-	-
50	1.005	8.7	-	-
15-50	-	-	-	0.10

* (U) Measured by the DuNouy ring method.

** (U) Measured by the cloud-point method in a modified Beckmann freezing-point apparatus.

3. (C) VE (EA 1517).

(C) About 20 samples of VE were received during the period 23 April 1954 to 9 December 1955. Only cloud points in water were determined with several of the samples (table 4), in order to test the effectiveness of various purifying measures. Some of the samples were so impure that the measurement of additional physical properties was unwarranted. More extensive water-solubility data have been reported elsewhere.* The flash point of an impure sample not listed in table 4 was found to be 157°C. Intermittent efforts to freeze VE were made during a period of 1 yr. Previous work has shown that crystallization may be induced in organic liquids by cooling to vitrifaction at -196°C in liquid nitrogen, followed by storage at temperatures from 5° to 10°C below the melting point. Crystallization may also be accomplished by seeding a supercooled liquid with crystals of a compound having a similar molecular structure. Vitrifaction of VE followed by storage at temperatures raised in 5°C increments from -71° to -13°C, and inoculation with crystals of VG and EA 1599 (the P = S isomer of VG) did not result in crystallization of VE.

* (U) Callahan, J. J., Coulter, P. B., and Zeffert, B. M. CWLR 2074. Solubility Studies With VE and VG. October 1956.

(C)

TABLE 4

PHYSICAL PROPERTIES OF VE (EA 1517) SAMPLES (U)

Sample	Density at various temp			Viscosity at various temp			Surface tension at various temp			Lower consolute temp in water
	25°C	35°C	50°C	25°C	35°C	50°C	25°C	35°C	50°C	
	g/ml			cs			dynes/cm			°C
23 Apr 54	1.0318	1.0220	1.0095	7.32	5.30	3.08	-	-	-	-
2884	1.0162	1.0075	0.9948	5.53	4.16	2.90	30.7*	-	-	ca. 37
2906	1.0176	1.0090	0.9959	5.62	4.19	2.90	-	-	-	ca. 39.5
3100	-	-	-	-	-	-	-	-	-	41.3
PB no. 4	1.0180	1.0094	0.9965	5.44	4.08	2.85	29.5**	28.9**	27.3**	40.6
9003	-	-	-	-	-	-	-	-	-	41.4

* (U) Measured by the DuNouy ring method.

** (U) Measured by the capillary-rise method.

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A 5-ml sam[illegible] ity was measured. A value of 0.995 g/m. was obtained at 25.[illegible]

6. (C) EA 1576.

One sample of EA [illegible] was received. The properties measured are shown in table 5.

(C) TABLE 5

PHYSICAL PROPERTIES OF EA 1576 (ANAL. 23[illegible]6-A[illegible])

Temperature	Density	Viscosity	Surface tension
°C	g/ml	cs	dynes/cm
25	1.0829	23.3	32.[illegible]
35	1.0743	14.7	-
50	1.0616	8.38	-

(U) Measured by the DuNouy ring method.

6. (C) EA 1622.

(C) A single sample of compound EA 1622 was received for physical properties measured are shown in table 6. Subsequent samples will probably be purer and may yield different values. The same vitrification and storage treatment applied to VE was given to EA 1622 inoculation was carried out and no crystallization took place.

(C) TABLE 6

PHYSICAL PROPERTIES OF EA 1622 (SAMPLE 3098)[illegible]

Temperature	Density	Viscosity	Surface tension *
°C	g/ml	cs	dynes/cm
25	1.023	6.10	29.
35	1.014	4.49	29.[illegible]
50	1.000	3.06	28
Flash point:**	135°C		

* (U) Measured with the Cassel tensiometer

** (U) Determined in a semimicro apparatus.

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7. (C) VM (EA 1664).

(C) Only two of the five samples of VM received were in sufficient quantity to permit more than cloud-point determinations in water and surface-tension measurements with the Cassel tensiometer. VM was given the same vitrifaction and storage treatment as VE but it did not freeze. Table 7 lists the values of physical properties obtained in these Laboratories.

(C) TABLE 7

PHYSICAL PROPERTIES OF VM (EA 1664) SAMPLES (U)

Sample no.	Density at various temp			Viscosity at various temp			Surface tension at various temp		
	25°C	35°C	50°C	25°C	35°C	50°C	25°C	35°C	50°C
	g/ml			cs			dynes/cm		
2860-b	1.0280	1.0192	1.0064	5.77	4.32	3.00	31.0 a/	-	-
3051, fraction 2	-	-	-	-	-	-	-	-	-
3051, fraction 3	1.0311	1.0225	1.0095	5.85	4.33	2.99	31.1 b/ 30.7	30.5 b/ 29.8	28.5 b/ 28.6
9002	-	-	-	-	-	-	31.2 c/	-	-
9012	-	-	-	-	-	-	30.9 c/	30.1 c/	29.0 c/

a/ (U) Measured by the DuNouy ring method.

b/ (U) Measured by the capillary-rise method; second value obtained 2 mo later; densities identical for the same time period.

c/ (U) Measured with the Cassel tensiometer.

8. (C) VS (EA 1677).

Four samples of VS were obtained, the last two in too small a quantity for cloud-point determinations in water for aid in the assessment of purification procedures. Sample no. 3089 froze after 2 wk of daily vitrifaction in liquid nitrogen and storage at -70°C. An approximate melting point of -35°C was observed. The physical properties which was measured are shown in table 8. Water-solubility data for sample 9011, fraction 13, are given in table 9 and figure 1. The mixture froze in the lower-agent concentrations making cloud-point determinations impossible.

(C)

TABLE 8

PHYSICAL PROPERTIES VS (EA 1677) SAMPLES (U)

Sample no.	Density at various temp			Viscosity at various temp			Surface tension* at various temp			Lower consolute temp in water	Flash point**
	25°C	35°C	50°C	25°C	35°C	50°C	25°C	35°C	50°C		
	g/ml			cs			dynes/cm			°C	°C
3089	1.0000	0.9919	0.9795	-	-	-	-	-	-	-	-
9004	1.0016	0.9936	0.9817	9.36	6.64	4.37	30.2	29.3	28.1	-	-
9011, fraction 12	-	-	-	-	-	-	-	-	-	-	-
9011, fraction 13	-	-	-	-	-	-	29.9	29.2	27.9	ca. -5	168

* (U) Measured with the Cassel tensiometer.

** (U) Values determined in a semimicro apparatus.

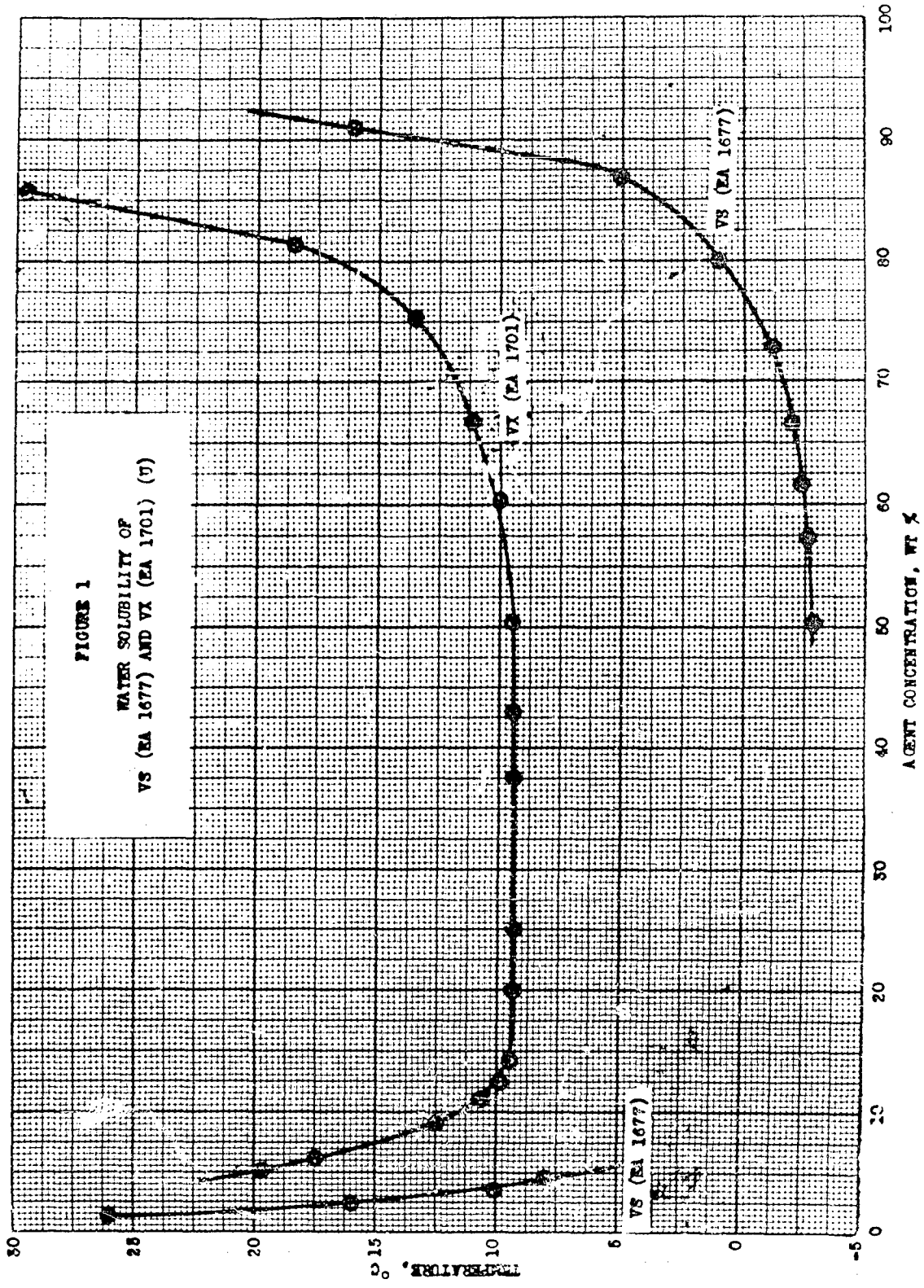


FIGURE 1

WATER SOLUBILITY OF
VS (EA 1677) AND VX (EA 1701) (U)

(C) TABLE 9

WATER SOLUBILITY* OF VS (EA 1677)
(ANAL. 9011, FRACTION 13) (U)

Agent concn	Cloud point	Agent concn	Cloud point
wt %	°C	wt %	°C
1.4	26.	61.6	-2.3
2.7	16.	66.8	-1.9
3.7	10.	72.8	-1.2
4.8	8.	80.0	1.0
28.6	-4.7	86.9	5.
50.1	-2.9	90.9	16.
57.2	-2.6		

* (U) Measured by the cloud-point method in a modified Beckmann freezing-point apparatus.

9. (C) EA 1694.

Only one sample of EA 1694 was submitted after purification in the molecular still at 80°C and 5 x 10^{-4} mm Hg pressure. The physical properties measured are shown in table 10. The same vitrifaction and warming treatment given VE did not result in crystallization of this sample. The EA 1694 was miscible with water over the temperature range from 0° to 100°C.

10. (C) EA 1699.

Only one sample of this material was received, purification was accomplished in a molecular still at a pressure of 5 x 10^{-4} mm Hg and temperature of 80°C. Physical properties are listed in table 11.

(C) TABLE 10

PHYSICAL PROPERTIES OF EA 1694 (SAMPLE W) (U)

Temperature	Density	Viscosity	Surface tension
°C	g/ml	cs	dynes/cm
25	1.0453	4.92	30.8*
			31.5
			31.65**
35	1.0367	3.70	30.3*
			30.4
50	1.0236	2.61	29.2*
			29.3

* (U) Measured by the capillary-rise method; second value 2 mo later.

** (U) Measured with the Cassel tensiometer.

(C) TABLE 11

PHYSICAL PROPERTIES OF EA 1699 (SAMPLE 3034) (U)

Temperature	Density	Viscosity	Surface tension
°C	g/ml	cs	dynes/cm
25	1.0600	5.31	31.4*
			32.0
			32.7**
35	1.0510	3.96	30.4*
			31.0
50	1.0379	2.74	29.1*
			29.8

* (U) Measured by the capillary-rise method; second value 2 mo later.

** (U) Measured with the Cassel tensiometer.

11. (C) VX (EA 1701).

Seven samples of VX were received after various treatments in these Laboratories. One of the samples was inoculated with crystals of VS and the VX appeared to freeze slowly during several weeks of storage in a sealed container at -60°C. A good melting point was not obtained, although the sample appeared clear and liquified at -39°C. Subsequent storage of the molten material at -45°C for 2 mo with intermittent vitrifaction in liquid nitrogen, and seeding with crystals of VS, as well as EA 1728, did not result in crystallization. A sample of VX stirred at -60°C and seeded with material from the apparently solid VX did not show any crystalline growth. Such growth would have been slowed down considerably by the high viscosity of the liquid at -60°C. Physical properties of the several samples received are reported in table 12. Water-solubility data for sample 9000 are given in table 13, and plotted in figure 1.

(C)

TABLE 12

PHYSICAL PROPERTIES OF VX (EA 1701) SAMPLES (U)

Sample	Density at various temp			Viscosity at various temp			Surface tension at various temp			Lower consolute temp in water	Flash point
	25°C	35°C	50°C	25°C	35°C	50°C	25°C	35°C	50°C		
	g/ml			cs			dynes/cm			°C	°C
3058	1.0040	0.9958	0.9834	9.47	6.68	4.36	28.9 a/	28.1 a/	26.7 a/	ca. -3	-
PB no. 2	1.0098	1.0017	0.9893	10.05	7.00	4.49	30.5 a/	29.5 a/	28.2 a/	ca. 4	-
3121	-	-	-	-	-	-	-	-	-	ca. 6	-
3132	-	-	-	-	-	-	-	-	-	ca. 4	-
9000	-	-	-	-	-	-	31.6 b/	29.6 b/	28.2 b/	9.4	159 c/
9005-3	1.0099	1.0017	0.9895	-	-	-	-	-	-	-	-
9009	1.0083	1.0001	0.9876	9.96	6.95	4.48	-	-	-	ca. 9	-

a/ (U) Measured by the capillary-rise method.

b/ (U) Measured with the Cassel tensiometer.

c/ (U) Determined in a semimicro apparatus.

(C) TABLE 13

WATER SOLUBILITY* OF VX (EA 1701) (ANAL. 9000) (U)

Agent concn	Cloud point	Agent concn	Cloud point
wt %	°C	wt %	°C
4.8	21.5	37.7	9.4
6.3	17.5	43.0	9.4
9.1	12.5	50.2	9.5
11.2	10.6	60.2	10.0
12.6	9.8	66.8	11.2
14.4	9.5	75.1	13.5
16.8	9.4	81.2	18.5
20.1	9.4	85.8	29.5
25.1	9.4		

* (U) Measured by the cloud-point method in a modified Beckmann freezing-point apparatus.

12. (C) EA 1728.

(C) Only one sample of compound EA 1728 was received for the measurement of physical properties which are shown in table 14.

(U) Water-solubility data for EA 1728 are given in table 15 and plotted in figure 2.

13. (C) EA 1763.

(U) One sample of EA 1763 (anal. 9167) was received; the physical properties measured are given in table 16. The critical portion of the EA 1763 and water coexistence curve, was completely obscured by the freezing of the water from the mixture. The remaining portion of the curve is shown in figure 2, data for which are given in table 17. The sharpness of the cloud points at the low-agent concentrations indicated the sample to be of high purity. Repeated vitrifaction in liquid nitrogen followed by storage at -70°C did not produce crystals.

(C) TABLE 14

PHYSICAL PROPERTIES OF EA [illegible]28
(ANAL. 9022, FRACTION 2) (U)

Temperature	Density	Viscosity	Surface tension a/
°C	g/ml	cs	dynes/cm
25	0.9899	11.4	29.2
35	0.9819	7.8	28.6
50	0.9699	4.9	27.2

L.C.T. b/, -1.6°C
Flash point c/, 170°C
mp, -12.1°C

a/ (U) Measured with the Cassel tensiometer.

b/ (U) Lower consolute temperature in water.

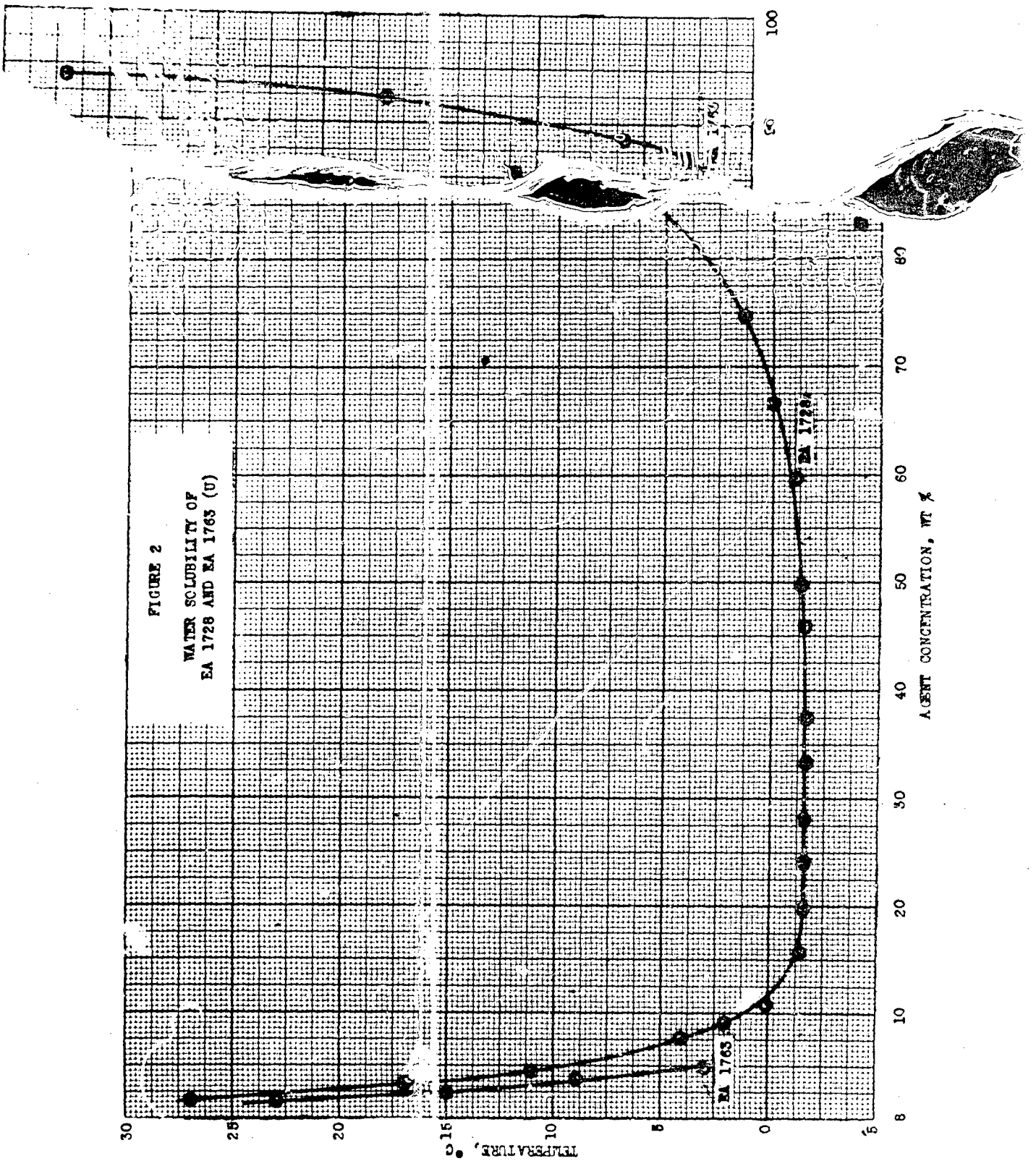
c/ (U) Values determined in a semimicro apparatus.

(C) TABLE 15

WATER SOLUBILITY* OF EA [illegible]28 (U)

Agent concn	Cloud point	Agent concn	Cloud point
wt %	°C	wt %	°C
1.6	27.	27.2	-1.6
3.2	17.	33.2	-1.7
4.3	11.	37.4	-1.6
7.6	4.	46.0	-1.5
9.0	2.2	49.8	-1.4
10.7	0.2	59.9	-1.0
15.7	-1.4	66.5	0.
18.7	-1.5	74.9	1.5
23.0	-1.6	85.6	6.0

* (U) Measured by the cloud-point method in a modified Beckmann freezing-point apparatus.



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(U) TABLE 16

PHYSICAL PROPERTIES OF EA 1763 (ANAL. 9167) (U)

Temp	Viscosity	Surface Tension *
°C	cs	dynes/cm
25	[illegible]	30.4
35	0.[illegible]	29.3
55	0.9[illegible]	28.1

L.C.T., ** -0°C (see table [illegible])

* (U) Measured by the cap[illegible]

** (U) Lower consolute temp[illegible]

(C) TABLE 17

WATER SOLUBILITY * OF EA [illegible]
(ANAL. 9167) (U)

Agent concn	Cloud point	Agent concn	Cl[illegible] pt
wt %	°C	wt %	°C
1.6	23	87.0	-[illegible]
2.4	15	88.9	[illegible]
3.7	9	92.3	12
4.8	3	94.1	27
83.3	-4		

* (U) Measured by the cloud-point method in a modified Beckmann freezing-point apparatus.

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